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J. A. KRUG, SECRETARY

BUREAU OF MINES
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POTT-BROCHE COAL-EXTRACTION PROCESS AND PLANT OF RUHRÖL G.M.B.H., BOTTROP-WELHEIM, GERMANY

BY

H. H. LOWRY AND H. J. ROSE

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POTT-BROCHE COAL-EXTRACTION PROCESS AND PLANT OF RUHRÖL G.m.b.H., BOTTROP-WELHEIM, GERMANY[1]

By H. H. Lowry[2] and H. J. Rose[3]

FOREWORD

This report is one of a series written by members of the Solid Fuels Mission to Germany describing wartime developments in the mining, preparation, and utilization of coal. This mission was organized early in 1945 under the auspices of the Technical Industrial Intelligence Committee, which cooperated with a counterpart British committee. The Solid Fuels Mission operated under the direction of a steering committee headed by Dr. C. J. Potter, Deputy Solid Fuels Administrator. The personnel of the mission consisted of J. W. Buch, Thomas Fraser, L. L. Newman, L. D. Schmidt, and H. F. Yancey of the Bureau of Mines; H. H. Lowry, Director of the Coal Research Laboratory, Carnegie Institute of Technology; H. J. Rose, Vice-president and Director of Research, Bituminous Coal Research, Inc.; and F. H. Reed, Chief Chemist, Illinois Geological Survey.

Over 50 reports were written by the members of the Solid Fuels Mission. These, together with microfilms of supporting data obtained in the course of the investigations, are on file in the Bureau of Mines, Washington, D. C., where they are available for public examination. The Office of the Publication Board in the Department of Commerce is prepared to furnish photolith copies, photostats, or microfilm reproductions of all declassified reports. A Bibliography of Scientific and Industrial Reports is issued weekly by the Department of Commerce, Office of the Publication Board, and is sold by the Superintendent of Documents, Government Printing Office, Washington 25, D. C., on a subscription basis. An initial payment of $10 will provide for approximately 9 months, and subscribers will be notified when additional remittance is necessary. All subscriptions will begin with volume 1, No. 1, unless ordered otherwise. The Bibliography covers all materials and subjects and gives instructions for obtaining the desired items.

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[1] The Bureau of Mines will welcome reprinting of this paper provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Information Circular 7420."
Prepared from Combined Intelligence Objectives Subcommittee report "Coal Extraction Plant of Ruhröl G.m.b.H. at the Hugo Stinnes Werke of Mathias Stinnes Gewerkschaft in Welheim Near Bottrop i. W.", by H. H. Lowry and H. J. Rose. 1945. (C.I.O.S. No. XXXI-27, Item No. 30; Solid Fuels No. 21.) Appendix prepared from C.I.O.S. report "Visit to Botrop-Welheim Hydrogenation Plant," by C. Cockram. 1945. (C.I.O.S. No. XXX-104, Item No. 30.)

[2] Director, Coal Research Laboratory, Carnegie Institute of Technology.

[3] Vice-president and Director of Research, Bituminous Coal Research, Inc.

The reports of the Solid Fuels Mission relate to the following general subjects: Mining methods in the Ruhr district of Germany, with special reference to mechanical cutting and loading equipment, pneumatic packing, and Diesel mine locomotives; activities of industrial research laboratories supported by the German coal and coke industry; coal preparation in western Europe; low-temperature carbonization processes; metallurgical coke from weakly coking coals; low-ash electrode carbon from coal; new developments in the production of metallurgical coke and the utilization of gas and byproducts; briquetting brown coal and bituminous coal without the use of binders; recent engineering developments in gas turbines and steam generators, with special reference to use of coal; German coal-fired heating and cooking stoves; and the commercial extraction of coal with solvents for production of synthetic liquid fuels or chemical products.

Numerous reports on the activities of German synthetic liquid fuels plants and the plants for the production of synthesis gas from solid fuels are available in the same office as the solid fuels reports.

It is planned to publish in this series of Bureau of Mines Information Circulars the more important reports or combination of reports prepared by the members of the Solid Fuels Mission. A similar series on synthetic fuel activities is being prepared.

A. C. FIELDNER, Chief,
Fuels and Explosives Branch.

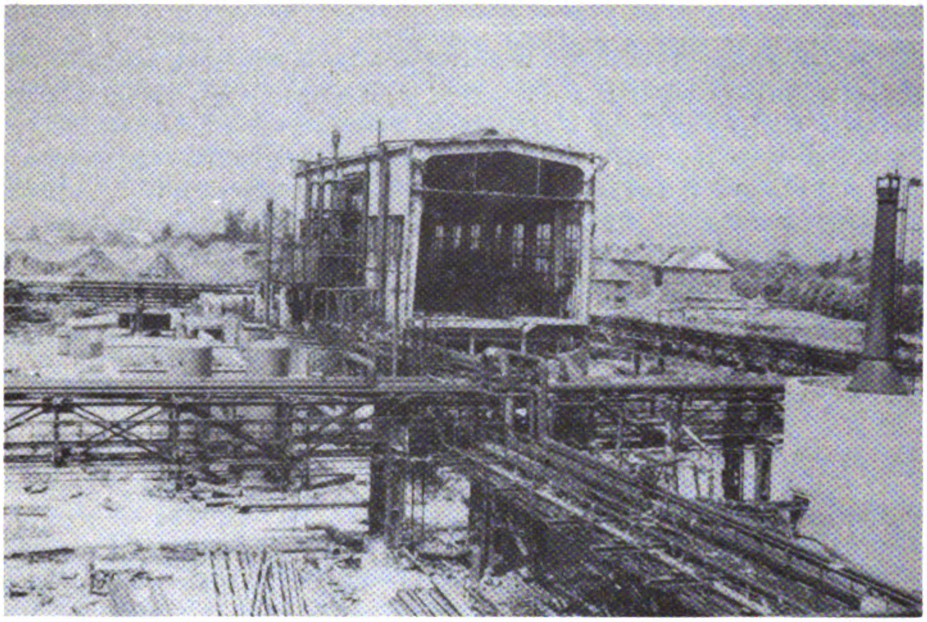

Figure 1. - End view of filter building, Pott-Broche coal-extraction process, and overhead plant piping, Ruhröl G.m.b.H., Welheim, Germany, June 30, 1945. (Photo by H. J. Rose. Neg. 213-8.)

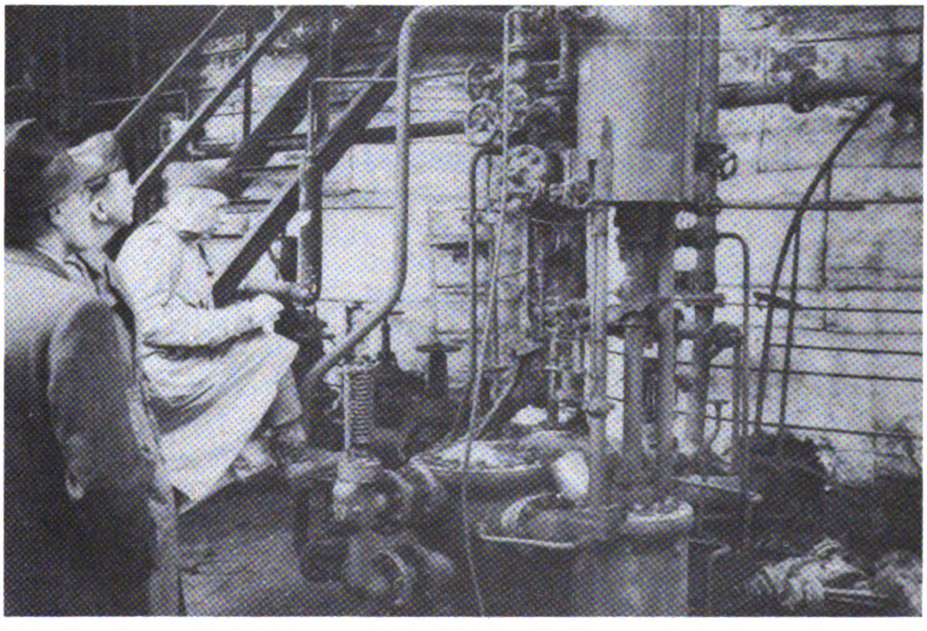

Figure 3. - Mixing kettles for coal and oil and pumps, Pott-Broche coal-extraction process, Ruhröl G.m.b.H., Welheim, Germany, June 30, 1945. (Photo by H. J. Rose. Neg. 212-8.)

INTRODUCTION

The Pott-Broche process for extracting coal with solvents at elevated temperatures was originally intended for use in obtaining an ash-free or low-ash mixture that could be hydrogenated readily to liquid hydrocarbons. In practice it was found more desirable to distill the solvent and certain oils from the extract. The residue from distillation was then carbonized to produce a very low-ash electrode carbon. This report, prepared after an inspection of the plant for the U. S. Technical Industrial Intelligence Committee, is intended to supply information on plant operation in Germany. Authors of several chapters of Chemistry of Coal Utilization[4] have discussed the technical aspects of the extraction process and have listed references to the original material.

CONCLUSIONS

The extraction process is feasible mechanically. It is uncertain that, even in Germany, the process could be justified without having available the special solvent oils from hydrogenation of pitch. One of the major advances in the technical operation of the process during the war was a new filtering system employing specially developed ceramic filters of high porosity, which permitted recovery of an extract containing only 0.05 percent ash. The filters and filtering system may have other industrial uses.

The Plant and Process

The coal-extraction plant (fig. 1) is in Welheim, near Bottrop, in Westphalia, and is associated with a hydrogenation plant, with which it is integrated. The plant occupies an area of 350,000 square meters (86.5 acres) and was first bombed on July 21, 1944. There were 10 raids, the heaviest damage being done on October 31, 1944, since which the plant has not operated. Most of the coal-extraction equipment could be salvaged, but it is highly improbable that it could be operated without starting up the hydrogenation plant, which was largely destroyed.

The Pott-Broche process has been under development for many years, although only one plant has been built. This plant was built in 1937, but it was not operated continuously. At first there were many difficulties. It was the original intention to hydrogenate the coal extract to produce fuel oil, but it was found that the capacity of the hydrogenation plant was greater when operating on coal-tar pitch (of which there was a large surplus in wartime Germany) than on coal extract. Consequently, the coal-extraction plant was shut down for some time. Then it was discovered that the middle oil from the hydrogenation of pitch was an excellent solvent for use in coal extraction, and it was used instead of the original tetralin-cresol mixture. Furthermore, after use in the coal-extraction process, the recovered middle oil was better for vapor-phase hydrogenation, because the motor-fuel produced contained a higher amount of aromatics. The coal-extraction plant had been operating

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[4] National Research Council Committee, H. H. Lowry, editor, Chemistry of Coal Utilization: John Wiley & Sons, Inc., New York, 2 vols., 1,868 pp.

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since 1941-42 until it was bombed out late in 1944. The coal extract was coked for the manufacture of high-grade electrode carbon of exceptionally low (0.1 to 0.15 percent) ash content. Several important patents have been issued.[5]

Coals having more than 25 percent volatile matter (Muck method) are suitable for the process. Those of lower volatile-matter content yield less extract. Dr. Broche said that the process was also suitable for higher-oxygen bituminous coals like Saar coal, which gave as high yields as Ruhr coal. The bright components (clarit and vitrit) of bituminous coal are extracted well; the dull coal (durit) is extracted in part, whereas the fusain is not extractable. Brown coal is not suitable on account of the large amount of decomposition.

The original solvent was four parts of tetrahydronaphthalene and one part of a mixture of the three isomeric cresols boiling from 190° to 210°. This solvent was used with half its weight of coal as the mixture to be treated in the extractor. The same ratio of solvent to coal was used later, but the solvent was changed to a middle oil with a boiling range from 200° to 320°. C. and about 9 percent hydrogen, from the hydrogenation of coal-tar pitch. The pitch had a melting point of 67° and was obtained from tar produced by normal high-temperature carbonization of bituminous coal. It is important that the solvent contain hydronaphthenes with disposable hydrogen as furnished by the tetralin in the original solvent. The tetralin originally used was reduced to naphthalene in the process, and the naphthalene had to be recovered and regenerated for economical operation. The regeneration was the stage that ruled out this solvent, as the sulfur in the recovered liquid product poisoned the Ni catalyst used for hydrogenation.

Earlier papers on the process emphasized the necessity of a definitely predetermined and controlled temperature schedule in the process. It has now been determined that this is unnecessary and that it is only important that the maximum temperature does not exceed the decomposition temperature of the insoluble residue (Restkohle). This temperature differs for different coals, and for Stinnes coal of 28 percent volatile, on a dry, ash-free basis, it is 410°. A higher temperature, such as 440° to 450° C., would give much trouble from coke formation in the tubes. This coal would begin to yield tar at 350° C. when heated under ordinary conditions. Broche stated that early work indicated the need for using a high enough temperature. For example, only part of a coal may be extracted at 380°, with increasing amounts at 390° and 400°, but still more complete extraction at 410° C., in which case the last temperature would be used. Extracts made at too high or too low a temperature would be hard to filter. The optimum temperature for processing a particular coal would be determined by laboratory extraction tests at a series of temperatures. Broche referred to the process as the "Aufschluss," i. e. the decomposition or "unlocking" of coal. In reply to questions, he said that he did not know how to dissolve a large percentage of coal without decomposing it, nor did he know how to extract a large percentage while working at atmospheric pressure. The

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[5] German patents 633,391; 633,693; 663,497; 687,898; 691,529. British patents 462,478; 464,337; 480,214; 480,644; and U. S. patents 2,123,380; 2,241,615.

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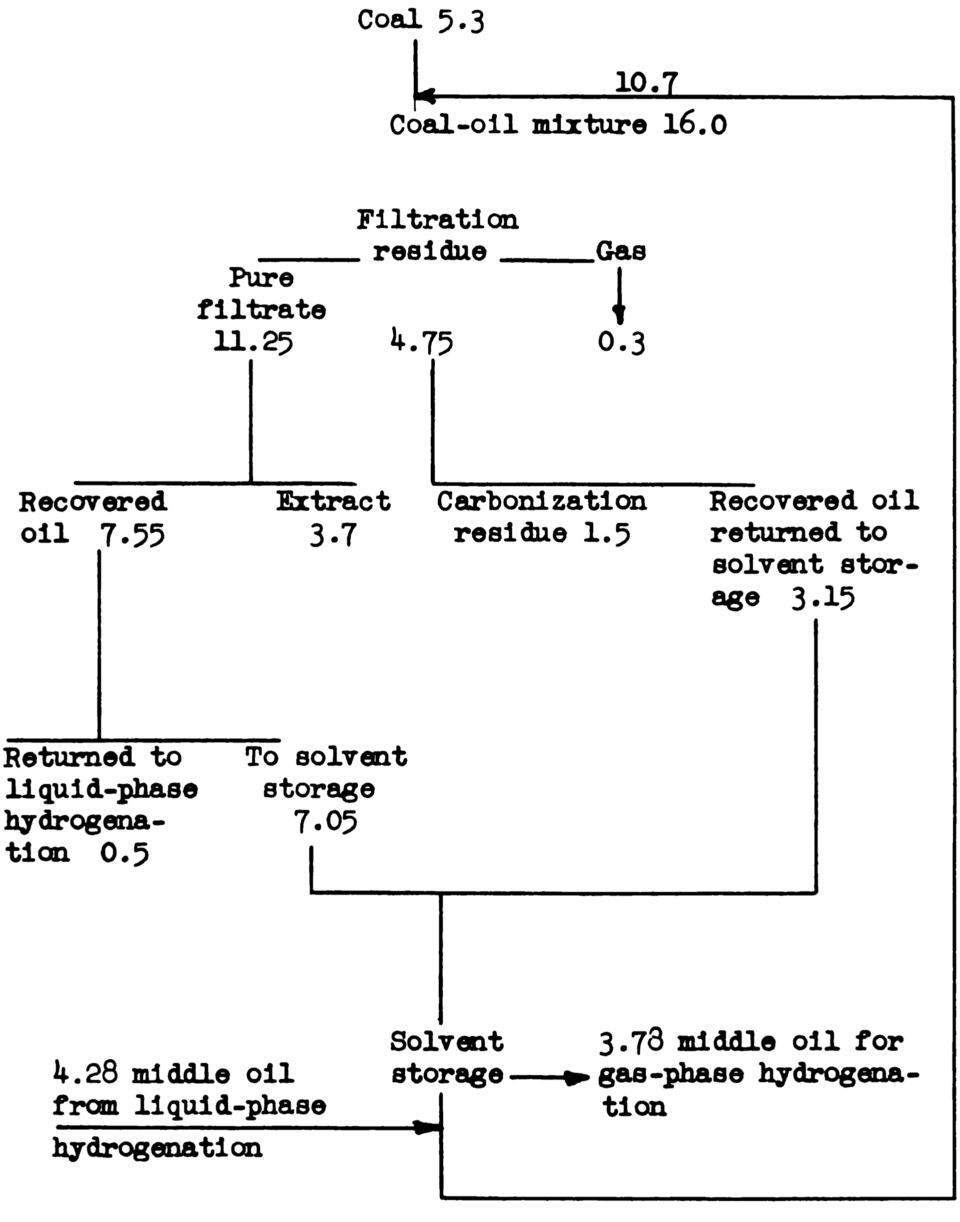
Coal 5.3
10.7
Coal-oil mixture 16.0
Filtration residue
Gas
Pure filtrate 11.25
4.75
0.3
Recovered oil 7.55
Extract 3.7
Carbonization residue 1.5
Recovered oil returned to solvent storage 3.15
Returned to liquid-phase hydrogenation 0.5
To solvent storage 7.05
Solvent storage
3.78 middle oil for gas-phase hydrogenation
4.28 middle oil from liquid-phase hydrogenation

Figure 2. - Flow sheet of extraction process at Welheim.

maximum temperature and the vapor pressure of the oil determine the maximum pressure - which ranges from 100 to 150 atmospheres.

The following description was prepared from notes taken in the plant and from a flow sheet obtained at the plant and a print of a schematic layout of the preheater and extractor equipment. These two items have also been microfilmed on T.O.M. Reel 70 as No. 000895 near the end of the reel. The flow sheet is for an annual production of 26,000 tons of extract calculated on the basis of 7,200 hours per year and is reproduced in part in figure 2, in which the numbers are tons per hour. The figures do not balance exactly in every case.

The coal used in the process as outlined is from the Stinnes I/II and Stinnes III/IV mines with a volatile matter content, upon a dry, ash-free basis, of 27 to 28 percent. Upon the same basis, a typical elementary analysis is C, 86.7 percent; H, 5.1 percent; S, 1.2 percent; N, 1.6 percent; and O (by diff.), 5.4 percent. The raw coal has a water content of 5 to 10 percent and an ash content of 5 to 7 percent. It is bunkered and fed to air-swept Büttner ball mills, where it is both ground and dried by flue-gas to about 0.5 percent water content. The product is about 95 percent through 180 mesh (4,900 mesh per square centimeter) and 65 percent through 240 mesh (10,000 mesh per square centimeter). So far as extraction is concerned, the fineness of the coal is not very important - they could use 0 to 10 mm. size for example. However, in order to pump the mixture, they found it necessary to pulverize the coal. The pulverized coal is stored in bunkers and fed by volume (on a rotating plate) to agitated mixing kettles (fig. 3) as the solvent is added.

The mixing kettles are steam-heated, of 50 cubic meters capacity each, and are maintained at 60° to 100° C. This temperature is not critical. The mixing is a batch process, and four kettles are available. The coal concentration is determined in each batch before being transferred to the extraction tubes. A new batch is started about every 3 hours. The coal-oil mixture is taken from the kettles by Atlaswerke transfer pumps to high-pressure pumps (150 atm), each having 15 cubic meters per hour capacity, for introduction to the preheating and extraction tubes. The three high-pressure pumps had been destroyed but were said to have been of conventional design.

The preheating and extraction tubes are assembled in a fire-brick furnace heated by flue-gas circulation. The temperature of the gas is maintained at about 550° C., and the gas is only in direct contact with the preheater tubes, which at the entrance of the coal-oil mixture are at about 100° C. and at the end of the preheating zone about 430° C. The preheater tubes completely surround the extraction chamber itself, which is thus effectively thermostated. The material entering the extraction chamber is at about 430°, and it leaves at about 415° C., the drop in temperature being explained as heat necessary for the depolymerization reactions. The total time from entrance to preheater to outlet of extraction chamber is about 1 hour, 20 percent of the time being in the preheater. If a much shorter time of treatment were used, the product could not be filtered, because of insufficient depolymerization.

Both preheating (fig. 4) and extraction tubes are hairpins, or inverted "U"'s, about 17 meters high. The preheating tubes (fig. 5) are of uniform diameter and are Mannesmann finned tubes of N-8 steel, 70 mm. i.d., and 102 mm. o.d., designed for withstanding 300 atm. pressure, which is higher than necessary. Forty of these preheater tubes are shown in Ruhröl drawing BK 16-2. The extraction tubes have the down-leg 120 mm. i.d. and the up-leg 185 mm. i.d. and a wall thickness of 8 mm. The up-leg was made of larger diameter because of anticipated effects due to gas evolution, but this is not now believed necessary. As last operated, there were 24 hairpins in the extractor sections of the furnace. Originally, 48 extraction hairpins were provided, but experience showed that 24 were sufficient, and one reactor tube chamber was empty when observed. With 48 tubes, bad coking occurred in the last tubes. There appears to be an optimum time in the extractor section, too long a time causing excessive coking and too short a time giving a product not readily filtered. It is necessary to close down every 2 to 3 months and remove the extraction tubes for cleaning. Replacement by spare tubes takes 5 days. The fouled tubes are cleaned by a mechanical borer, as the coke formed on the inner surfaces is very hard. No sludge traps or boots at the bottom of the hairpins were considered necessary.

From the extraction chamber, the material goes to a vessel, where it is cooled by water to 160° to 170° C. and is discharged through a pressure-reducing valve, which drops the pressure from 100 atm. to 1 atm. The gas formed in the process is mixed with other gas in the plant. The gas is about 70 percent saturated hydrocarbons - mostly CH_4, with 20+ percent hydrogen, the remainder being CO, CO_2, N_2, and H_2S, the last being 1 to 2 grams per cubic meter. No ammonia appears in the gas. The product is stored in tanks at 150° C. ahead of the filters (fig. 6). There has been no trouble from sludge settling out in the pipe lines of the plant.

The filtering operation is one of the most important steps. The filters consisted of pressure vessels each containing 32 filter "candles" in a vertical position, with a filtering surface about 2 meters long. Each "candle" (fig. 7) consists of an assembly of coarsely porous ceramic rings put together with washers. The individual rings measure:

Outside diameter.....	122 mm.	(4 13/16 in.)
Inside diameter......	81 mm.	(3 3/16 in.)
Wall thickness.......	20 mm.	(13/16 in.)
Height of ring.......	32 mm.	(1 1/4 in.)

and were said to have been manufactured from quartzite material by Schuhmacher Fabrik at Biettigheim, near Stuttgart. Dr. Broche did not know the details of manufacture, which were said to be a trade secret of Schuhmacher. Each ring was reinforced by two external welded wire bands. Experimental filter rings of more finely grained ceramic material with a corrugated external surface and about 8 inches high were seen in the plant. These were said to have been less satisfactory.

Each filter vessel with 32 candles provides 22 square meters of surface and was said to have a throughput of about 16 cubic meters (or tons) of liquid per hour, which is equivalent to about 90 tons of recovered solid extract per

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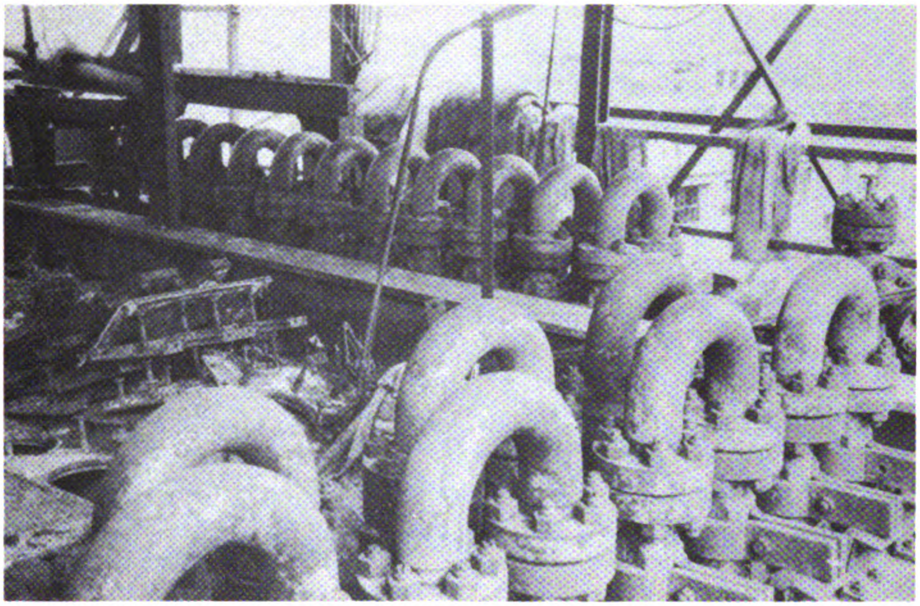

Figure 4. - Top of preheating tubes, Pott-Broche coal-extraction process, Ruhröl G.m.b.H., Welheim, Germany. (Photo by H. J. Rose. Neg. 213-7.)

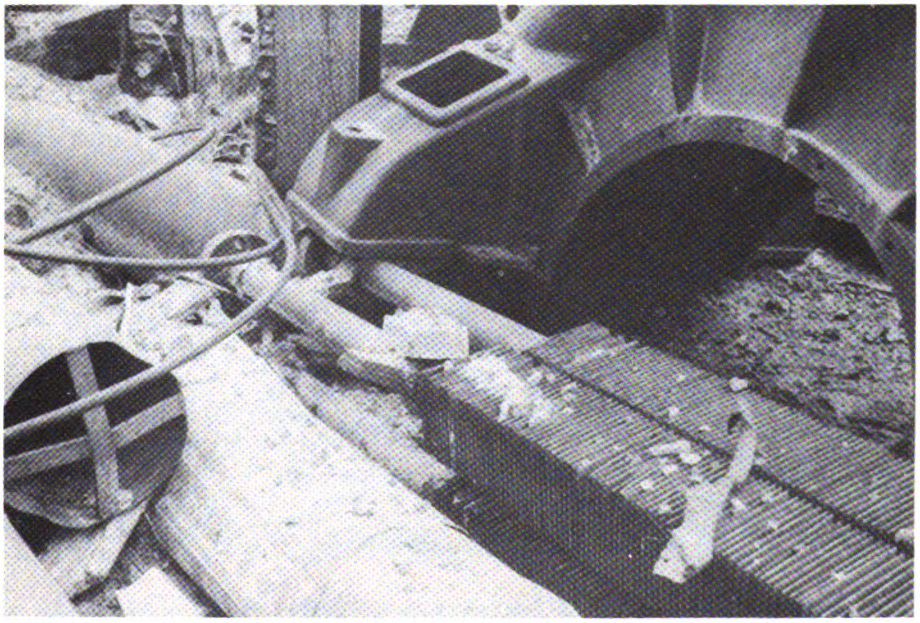

Figure 5. - Finned preheating tubes as used in Pott-Broche coal-extraction process, Ruhröl G.m.b.H., Welheim, Germany, June 30, 1945. (Photo by H. J. Rose. Neg. 213-5.)

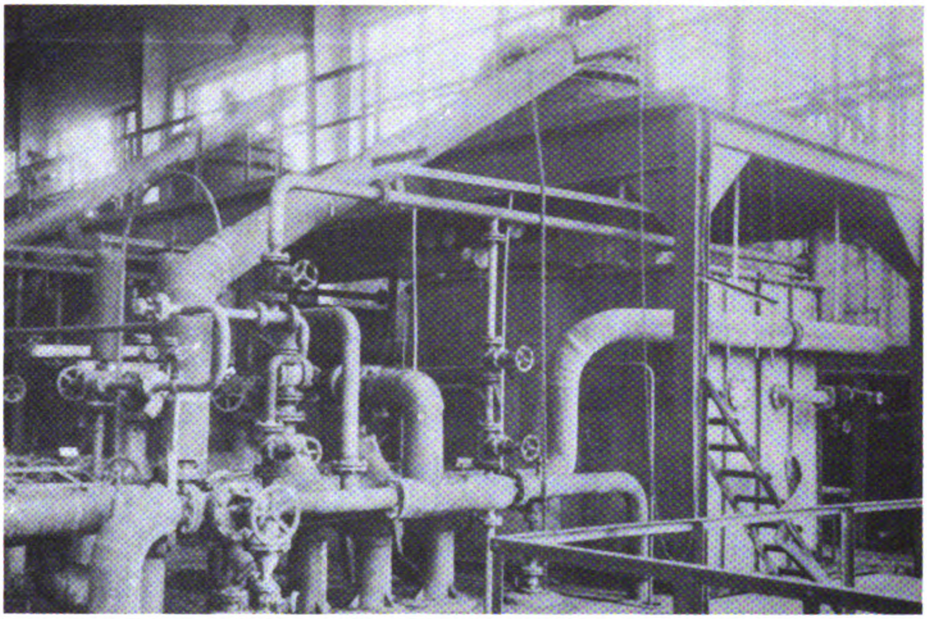

Figure 6. - Filter vessels and piping, Pott-Broche coal-extraction process, Ruhröl G.m.b.H., Welheim, Germany, June 30, 1945. (Photo by H. J. Rose, Neg. 214-3.)

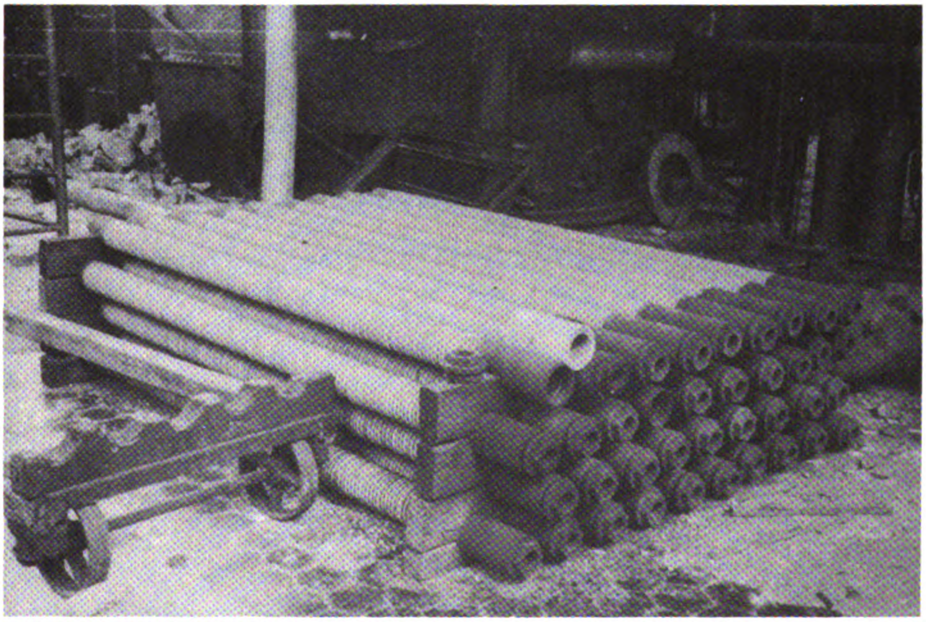

Figure 7. - Ceramic filter rings reinforced with wire bands and assembled into "candles." One unit of experimental ceramic filter about 8 inches long is shown on top of pile near center of picture. Pott-Broche coal-extraction process, Ruhröl G.m.b.H., Welheim, Germany, June 30, 1945. (Photo by H. J. Rose. Neg. 214-1.)

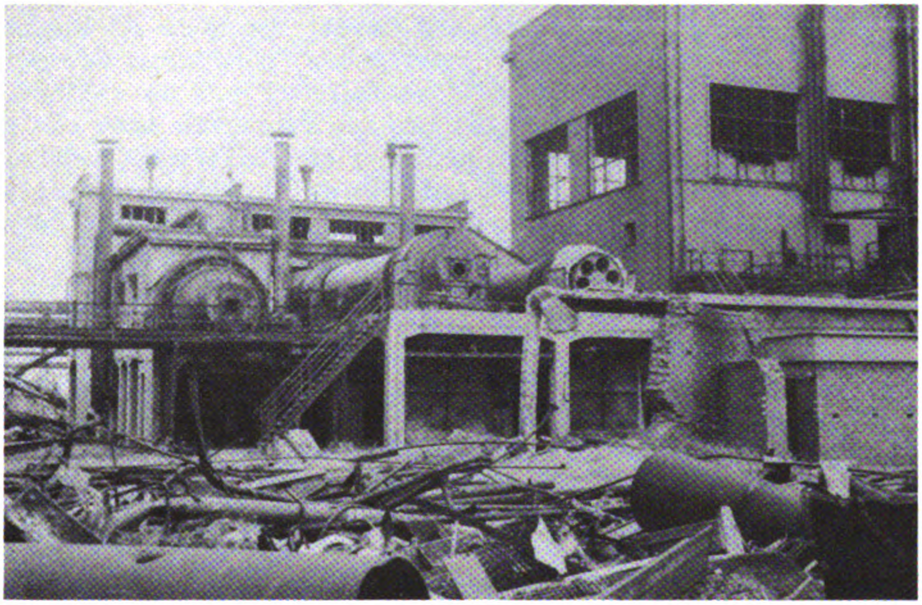

Figure 8. - Rotary kilns for de-oiling "Restkohle" (insoluble residue), Pott-Broche coal-extraction process, Ruhröl G.m.b.H., Welheim, Germany, June 30, 1945. (Photo by H. J. Rose. Neg. 214-6.)

24 hours. The plant had three filters of this type, any one of which was sufficient to handle the whole plant output. There was also an experimental filter of different design. The life of the ceramic filter rings was about 4,000 filtrations, which is equivalent to 1,000 hours, or 2 months, of continuous operation. They could be regenerated to some extent by washing and scraping (but not by burning), but it was preferred to replace them with new rings.

The solution of coal is forced around the outside of the "candles," the pressure being about 4 to 6 atm. and the temperature about 150° C., at a rate about 10 percent greater than the rate of filtration, so that there is some recirculation to storage. The throughput per filter is 16 cubic meters per hour. When the rate of filtration becomes low, cleaning is necessary, and this occurs 3 to 4 times an hour. The material in the pressure vessel is withdrawn and returned to storage. Fresh solvent at 150° is passed through the sludge layer on the outside of the filter, and the sludge is dried to a shell on the outer surface of the "candle" by CO_2 supplied at ordinary temperature and traveling the same direction as the solution being filtered. The cake of "Restkohle" is then broken off by a sudden increase in pressure of CO_2 to 6 or 7 atm. inside the candle. The residue falls into the conical bottom and is then transferred by conveyor to the rotary kilns (fig. 8) for distillation. As constructed, the filtering station has a capacity several times greater than the earlier stages of the process.

The three rotary kilns provided also have excess capacity. The largest unit has a capacity for treating the residue from 100 tons of coal per day for recovery of the oil, and there are two smaller kilns. The kilns contain 6 tubes 350 or 500 mm. in diameter and 12 m. long. The tubes are heated by flue gas, so that the final temperature of the "char" is 350° C. There are chains in the upper end of the tubes for breaking up the char. As discharged, the powdered char has about 0.2 percent oil content, an ash content of about 30 percent, and is used for boiler fuel.

The filtered solution - "reinfiltrat" - is stored in tanks at 150° and kept agitated. (If cooled to 20° C., it could still be stirred. Dr. Broche did not know of any possible commercial use for this filtrate, which contains both the coal extract and solvent oil.) From the storage tanks the filtrate goes to stills for separation of the solvent from the extract. The filtrate is first heated in a pipe still to about 250° C. at 10 to 12 atm. and is sprayed into a vacuum chamber having 40 to 50 mm. pressure, where the extract is concentrated from 25 to 50 percent content of solids. It leaves the vacuum column at about 200° and is reheated in a pipe still to about 350° to 360° at 10 to 15 atm., and then goes to the final distillation in a vacuum column at 30 mm. pressure. The extract discharged from the bottom of the vacuum column at 300° is said to be completely oil-free and as fluid as water at this temperature. It is discharged through an electrically heated outlet onto a steel conveyor belt, where water cooling may be employed.

The recovered oil is redistilled, a small residue being returned to the liquid-phase hydrogenation plant. This redistilled oil is about 1 or 2 percent greater in amount than the solvent used, owing to some distillation of the coal during the extraction stage, which more than balances oil losses in the process. The recovered solvent is a better raw material for vapor-phase hydrogenation for gasoline than the original solvent. As shown in the flow sheet, a part of the recovered solvent is returned to the hydrogenation plant and replaced by fresh middle oil. This is necessary to maintain an adequate supply of disposable hydrogen in the solvent. If the same oil were re-used four or five times, it would no longer work well. Dr. Broche recommends this coal-extraction process only in connection with a hydrogenation plant.

The coal extract has a volatile-matter content of more than 40 percent (the exact percentage is hard to determine because of foaming) and a melting point of 210° to 220° C. (This softening temperature is determined by placing a fragment in a capillary tube and heating in a liquid bath. The softening temperature is not sharply defined). The extract has 0.2 to 0.3 percent more hydrogen than the original coal, this hydrogen having been obtained from the middle oil used as solvent. The oxygen content has been reduced several percent. Analyses have shown the following ranges: C, 88.50 to 89.90 percent; H, 5.20 to 5.30 percent; N, 1.50 percent; S, 1.00 to 1.10 percent; O (by diff.) 3.75 to 2.15 percent; and ash, 0.05 percent. It is 100 percent soluble in pyridine, cresol, and cresol-tetralin mixtures and 50 percent soluble in benzene. Recovery upon the basis of the starting coal is about 70 percent. When nitrated to destroy caking properties and to lower the ignition temperature, it gives a product suitable for carbon-dust Diesel engines. It was said to work very well for this purpose. However, the actual commercial use was to produce carbon for electrodes by high-temperature carbonization, in which it was charged to regular byproduct coke ovens as a solid. It was not necessary to melt it and spray it in, as in the carbonization of pitch.

The yield of coke or carbon is about 66 percent, based upon the extract. The ash content of the coke ranges from 0.10 to 0.15 percent, being lower than either petroleum coke or pitch coke (the latter having 0.4 to 0.5 percent ash). The carbon sold for 180 M per ton, whereas pitch coke sold for only 90 M per ton. (These figures were confirmed by the A. G. der Kohlenwertstoff-Verbände at Bochum, which is a sales organization). The raw coal costs 20 marks per ton. Dr. Broche stated that they figured the price could be reduced to less than 100 M per ton of coal extract before coking, for a plant with an annual capacity of 75,000 tons of extract. Most of the carbon was used for electrode manufacture for the aluminum and ferrous industries. However, it was stated that it was an "excellent graphitizing agent" for electric steel.

The cost of this first plant was stated to be excessive - seven to eight million marks - primarily because the capacities of the different stages were not completely integrated. For instance, three commercial filters were available, any one of which had enough capacity for the plant output; and three rotary kilns were provided, the largest of which was sufficient for the plant output. Some standby equipment was doubtless desirable.

Some additional data on the extraction process is given in the Appendix of this paper, which is taken from a report by C. Cockram on behalf of British Ministry of Fuel and Power and U. S. Technical and Industrial Intelligence Committee.

APPENDIX

Pott-Broche Extraction Process

By this process, coal was heated to 400°-420° C. with a solvent at 100 atm. pressure, the solution was filtered to remove the ash and undissolved coal, and the solvent was then removed by distillation. No gas was added to the coal paste.

The plant started up using tetralin as a solvent; part of the tetralin was dehydrogenated to naphthalene, which was rehydrogenated at 25 atm. pressure. This was the reason for adopting the scheme for CO removal by copper liquor at 3° C. and 25 atm. for the production of make-up hydrogen gas. It was found that middle oil containing 7 percent cresols could be used as the solvent, and it reduced the cost of the process.

It was the intention to hydrogenate the extract at 700 atm. pressure, but development of the process was slow and bituminous coal-tar pitch was used as an alternative feed stock for the hydrogenation plant. This proved to be a more satisfactory material than the coal extract, and operation of the Pott-Broche process was continued for the purpose of making high-quality electrode carbon of low ash content. Dr. Frese suggested that the refractory nature of the extract was due to the high temperatures used in the extraction process.

The coal was ground and mixed with middle oil in the ratio: 1 of coal to 2 of middle oil. This was pumped to the extraction system, which was built in the form of a square preheater with circulating flue gas. There were 40 vertical preheater tubes arranged around the outside of the heater and 48 soaking tubes in the center. The number of working tubes in the soaking section had been reduced to 24. The time of contact was stated to be 1 to 1-1/2 hours. The soaking section was referred to as the autoclave.

No gas was circulated with the paste, but some gas was made in the process. To prevent gas locking, the rising legs of the hairpins were 180 mm. diameter and the downcomers were 120 mm. diameter. The pressure was 110 inlet, 100 ats. exit at the start of a run, and the tubes were cleaned when the pressure drop increased to 40 ats. and the inlet pressure reached 150 ats. The temperature in the soaking section was 433° C. The feed rate was stated to be 16 m^3/hr. of paste.

The solution was filtered in batch filters containing ceramic elements (Kurtzen filters). There were three filters in use. A fourth, which was of different design and had a subsidiary rotary filter mounted on top, appeared to have been used for development work. The filters comprised a circular tank with a conical bottom fitted with a plough for removing the cake when it had been blown off. The filter elements (estimated to be 20 per filter) were inserted through the top cover. Each element consisted of a splined rod, over which were fitted the ceramic rings 4-3/4 inches o.d., 3-1/4 inches i.d., by 1-1/4 inches deep. The rings were separated by klingerit washers, and the whole assembly was bolted up solid. The length of ceramic elements was 79 inches. The ceramic elements looked like a coarse sandstone, but they were

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obviously moulded. Each ceramic had two iron wires spot-welded around it to reduce the chance of bursting while blowing off the cake which was formed on the outside. The filters had pressure gauges marked with a red operating line - feed 2 atm., middle oil for washing the cake 8 atm., CO_2 for blow back 6 atm. We were told that the normal feed pressure was 5 atm.

The solution was let down from the extraction unit and stored in lagged tanks, from which it was pumped to the filter tank. After building up the cake, the solution was replaced by middle oil to wash the cake. The washed cake was blown off with CO_2 and was removed by the ploughs onto a conveyor beneath for transport to two rotating retorts, where it was carbonised. The vent gases from the filters were scrubbed with cold middle oil to reduce the losses of middle oil as vapour. We were told that they could do 3 to 4 filtrations per hour and that the ceramic elements were discarded after 5,000 filtrations.

The recovery of oil from the residue was stated to be 90 to 95 percent, leaving 0.2 percent of extractable oil in the coke. The filtered solution was distilled, first under vacuum to remove middle oil and heavy oil together, and the distillate was refractionated to separate the heavy oil which was produced from the coal during extraction and which amounted to about 8 percent on the coal. Sixty percent of the middle oil was recycled to the process, and 40 percent was added to the vapour-phase stall feed. The make up middle oil was liquid-phase middle oil from pitch hydrogenation.

It was stated that it was not possible to extract more than 80 percent of the coal substance without getting into difficulty in the filtration stage and the extract was too hard. The extract normally produced had a melting point of 220° C. The extract was carbonised at the coke ovens and was stated to yield 65 to 70 percent coke; the rest was gas.

The plant was designed to treat 125 t/day of coal, but examination of the records indicated that this had not been achieved for any reasonable period.

Actual yields of extract taken from records for 1943 and 1944 are given below. Only in one month (May 1944) did they succeed in making extract at the rate of 20,000 tns. a year.

TABLE 1

	Coal treated, tns./month	Extract, tns./month	Yield, percent	Tns. coal per hour
December 1943...	2,239	1,494	66.8	3.0
January 1944....	3,250	1,679	51.6	4.37
February........	2,104	1,086	51.6	3.04
March...........	2,834	1,160	41.0	3.8
April...........	1,813	960	52.8	2.52
May.............	3,043	1,800	59.0	4.08
June............	1,594	707	44.3	2.2
Total......	16,877	8,886	Av. 52.7	

From these figures, it is clear that they had considerable difficulty in achieving flow-sheet outputs.

Some analyses of the coal used and the products obtained were noted.

	Coal	Extract	Residue
C	86.2-86.7	89.1-89.3	86.3
H	5.1- 5.3	5.4- 5.5	4.0
N	1.7- 1.8	1.9- 2.1	1.45
S	1.3- 1.5	0.8- 0.9	1.5
Cl	0.14-0.20	.08	0.45

Frese made a particular point about the low-ash content of the extract and that it made a coke superior to bituminous coal tar-pitch coke for this reason. He also said that it was blended off with pitch before coking. Some ash analyses were noted where the ash content was as high as 0.17 percent on the extract.

Frese also said that Prof. Wilke at Oppau had been experimenting with the extract as a fuel for pulverised coal motors. It had been possible to reduce the ignition point of the extract from 400° to 200° C. by the addition of NO and NO_2.

BIBLIOGRAPHY

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The reports and patents may be procured from the Office of Technical Services, U. S. Department of Commerce, Washington, D. C., under the "PB" or patent number given. The U. S. Technical Oil Mission reel 150 may be purchased from the Photoduplication Service, Library of Congress, Washington 25, D. C. The price is approximately 6 cents a foot of film.

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